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13. ABSTRACT (Maximum 200 words)

Shock loading and quasi-static loading has been examined in annealed high purity tantalum. Investigation with positron annihilation lifetime spectroscopy, microhardness testing, and optical microscopy shows increased dislocation density with increased true strain, but no significant increases with increased strain rate, aside from small degrees of dynamic recovery and twinning observed in shock loaded samples. The authors conclude that these results support the theory that deformation in tantalum is controlled by dislocation drag above the Peierls stress and that strain rate effects, which are clearly observed in most other materials, are suppressed.

Quench hardening in annealed high purity tantalum has also been investigated. Microhardness testing results show quench hardening after quenching from temperatures above 1100°C with cooling rates of approximately 200°C/min; these observations appear to be consistent with vacancy clustering. However, the temperatures required to produce quench hardening exceed the maximum estimates of temperatures achieved during shock loading. The authors conclude that quench hardening is not expected to have any significant effect on shock loading in tantalum.

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Positron Annihilation Lifetime Studies of Deformed and Shock Loaded Tantalum

Final Progress Report

February 26, 1999

STATEMENT OF PROBLEM STUDIED

The goal of this research project was to provide a characterization of shock loading in tantalum and identify the physical mechanisms that are responsible, as well as how these mechanisms compare to the quasi-static response. The investigation included positron annihilation lifetime spectroscopy, microhardness measurements, TEM, and optical microscopy. Transmission electron microscopy results from previously published studies were also incorporated into the investigation.

Positron annihilation lifetime spectroscopy (PALS) had not previously been applied to the characterization of shock loading in tantalum. A non-destructive testing technique which is highly sensitive to the localized average electron density of the material, PALS is an excellent tool for providing additional insight into the shock loading phenomenon. It was hoped that this technique would allow for an examination of the material that would make clear materials properties that had not been previously detected.

In an attempt to further characterize shock loading in tantalum, quench hardening was investigated and the results compared to those for shock loading. Quench hardening has been demonstrated and characterized for pure metals such as aluminum, copper, and gold. It has been explained by vacancy strengthening mechanisms with various analytical techniques including mechanical testing, resistivity measurements, Laue X-ray diffraction, transmission electron microscopy, and monovacancy formation energy calculations. Vacancy strengthening is generally accepted to occur either by the clustering of vacancies or by the condensation of vacancies along dislocation lines. If a similar mechanism could be identified in tantalum, it might provide significant implications for shock loading.

SUMMARY OF MOST IMPORTANT RESULTS

Annealed high purity (99.95%) tantalum was shock loaded and quasi-static loaded and analyzed with positron annihilation lifetime spectroscopy and microhardness testing. Shock loading was conducted in compression with a split Hopkinson bar at strain rates of 7000 per second. Quasi-static loading was conducted in compression with an Instron machine at strain rates of approximately 0.0003 per second. For each strain rate, tests were run to approximate true strains of approximately 30% and 50%. Positron lifetime data was collected and analyzed with the

Positron Iterative Fit program for samples compressed at each of the two strain rates and each of the two total strains. The results show a substantial increase in the mean lifetime for all samples compared to those in the as received condition. A general trend of increasing lifetime with increasing true strain is also observed; however, very little, if any, difference is found between shock loaded and quasi-static loaded samples. These results are verified with Knoop microhardness testing. A literature search of shock loaded tantalum shows substantial differences between the stress-strain curves for shock loaded versus quasi-static loaded tantalum, including the areas of dynamic recrystallization and recovery during shock loading.

Although differences are observed in the form of dynamic recovery and twinning in the shock loaded samples, positron annihilation lifetime and microhardness measurements indicate that the overall effects of the defect concentrations in each are nearly identical. The results from this study are consistent with the theory that deformation in tantalum is controlled by dislocation drag above the Peierls stress and that strain rate effects are suppressed by the dependence of the Peierls stress on the strain rate. In other words, as the strain rate increases, the Peierls stress (or the stress required to move a dislocation from one low energy position over one row of atoms to another low energy position) also increases which retards dislocation motion. For this reason, the increased number of dislocations which are activated by shock loading do not produce increased work hardening as compared to quasi-static loading.

Positron annihilation lifetime spectroscopy shows increased lifetimes with increased true strain, indicating an increased number of positron trapping sites within the lattice as a result of the deformation of both shock loaded and quasi-static loaded samples. Further, the trapping sites appear to be dislocations, even though a large dislocation density is presumed necessary in order to sufficiently perturb the local electron density to a degree that could be measured with PALS. Although sensitivity to dislocations is typically not expected with positron annihilation lifetime spectroscopy, the equilibrium vacancy concentrations of tantalum over the possible temperatures experienced during shock loading are too low to have a significant effect on the positron lifetimes. Dislocation intersections also appear not to affect the lifetimes, since transmission electron microscopy has shown shock-loaded tantalum to contain a larger concentration of dislocation cusps and tangles than quasi-static loaded tantalum. In the case of dislocations, however, TEM results have been published that shows the densities of screw dislocations to be very similar for shock loaded and quasi-static loaded tantalum. Further, large dislocation densities are expected in the samples studied due to the reported flattening in the stress-strain curves at lower strains which indicates a saturation of work hardening for both compression rates. Based on these results, the observed increases in positron lifetimes are concluded to correlate with increased dislocation density in the tantalum samples.

Microhardness measurements are found to agree with positron lifetime results in a general trend of increasing hardness with increased strain. However, the microhardness results also show a significantly decreased hardness for the shock loaded samples compared to quasi-static loaded samples. Based on the true stress versus true strain results for the shock loaded tantalum samples and optical microscopy, it is determined that dynamic recovery is responsible

for the observed softening. The fact that positrons are not found to be sensitive to this effect may indicate that dynamic recovery occurs non-uniformly within the samples, as observed in tantalum explosively formed penetrators.

Optical microscopy reveals specific features which clearly distinguished shock loading from quasi-static loading. The shock-loaded samples were consistently found to contain twins. The twins were generally serrated and have been reported to form during the initial stages of shock loading. The quasi-static loaded samples contain a small concentration of grains that show large numbers of slip lines. These observations indicate that quasi-static loaded tantalum is more likely to deform via the motion of planar arrays of dislocations than is shock loaded tantalum, in which slip lines are not observed. Therefore, it appears that deformation from shock loading occurs via the slip of large numbers of uniformly distributed dislocations over short distances and along various slip planes, while deformation from quasi-static loading occurs via the slip of a smaller number of dislocations over longer distances and along specific slip planes.

Annealed high purity tantalum was also subjected to various heat treatments and analyzed with positron annihilation lifetime spectroscopy and microhardness testing. Samples were rapidly cooled (at rates of approximately –200°C per minute) in a vacuum furnace and subsequently annealed at various annealing temperatures. Although the positron lifetime data is found to be essentially unchanged over the range of temperatures studied, a small increase in microhardness was found with samples annealed at temperatures over 1200°C. The microhardness increases are found to be strongly indicative of a thermally activated process, although the calculated enthalpy of formation is less than half of the value reported for monovacancy formation. The difference between these values is consistent with a vacancy clustering mechanism by which clusters of vacancies are able to significantly impede dislocation motion and thereby increase the microhardness of the samples, whereas monovacancies are not. Further, vacancy clustering explains how the very small vacancy concentrations for tantalum could cause significant strengthening. The positron lifetime results are in agreement with the fact that the calculated equilibrium vacancy concentrations for tantalum at up to 1500°C are still below reported threshold levels. For this reason, quench hardening is not expected to have any significant effect on shock loading in tantalum.

In an attempt to advance positron annihilation lifetime spectroscopy as an analytical tool, Positron Iterative Fit, the PALS analysis algorithm that was developed previously by the authors, was further demonstrated to be superior to other analysis programs. The PIF program is based on the chi-square goodness of fit test and is unique in that it allows for a statistical analysis of the goodness of the model which best fits the data, thereby establishing a fitting threshold by which good fits may be discerned from poor fits. A literature search over the last thirty years shows widely varying lifetimes in annealed copper with a general decreasing trend. Analysis of copper samples subjected to various annealing times shows that annealing has only a minimal effect on the positron lifetimes, supporting the theory that the other positron annihilation lifetime fitting routines have adversely affected the reported values in literature. This theory is consistent with the authors' work demonstrating the sensitivities of these fitting programs to required input variables that are neither defined by these programs nor experimentally measurable.

LIST OF ALL PUBLICATIONS AND TECHNICAL REPORTS

D.M. Stepp, A Chi-square Goodness of Fit Analysis of Positron Annihilation Lifetime Spectra, Master's thesis, Duke University, 1995.

D.M. Stepp, A Positron Annihilation Lifetime Study of Shock Loading in Tantalum, Ph.D. dissertation, Duke University, 1998.

D.M. Stepp, P.L. Jones, "Accurate Calibration of PALS Systems with $^{207}_{83}$Bi and $^{60}_{27}$Co," currently under preparation, *Nuclear Instruments and Methods in Physics Research – Section B: Beam Interactions with Materials and Atoms.*

D.M. Stepp, P.L. Jones, G.W. Pearsall, and A. Crowson, "Positron Iterative Fit: A Statistical Approach to PALS," under revision for publication, *Nuclear Instruments and Methods in Physics Research – Section B: Beam Interactions with Materials and Atoms.*

SCIENTIFIC PERSONNEL SHOWING ANY ADVANCED DEGREES EARNED

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